# OPTIMIZATION OF MILLING CONDITIONS AND SINTERING PROCESSES IN AL-CNT ADVANCED NANOCOMPOSITES

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# OPTIMIZATION OF MILLING CONDITIONS AND SINTERING PROCESSES IN AL-CNT ADVANCED NANOCOMPOSITES

by

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## ABSTRAK

Carbon nanotubes (CNTs) baru-baru ini telah muncul sebagai bahan dengan sifat-sifat yang istimewa. Penyelidik telah mengkaji penggunaannya sebagai penguatan dalam matriks polimer dan seramik. Disebabkan untuk menjangka kesulitan fabrikasi, maka kajian telah dilanjutkan dengan menggunakan CNTs untuk menguatkan matriks logam. Dalam projek ini, penggilingan bola planet ini digunakan untuk membubarkan 2%wt MWCNTs dalam serbuk aluminium (Al). Mekanikal gabungan (MA) digunakan untuk menghasilkan edaran homogen dari 2wt% CNTs dalam serbuk Al. Pengaruh waktu penggilingan (sehingga dengan 40 jam) kelajuan (200-250 rpm), saiz bola (10, 15 dan 20 mm) dan PCA (asid stearat, metanol) terhadap perkembangan morfologi dari serbuk Al telah dikaji. Metalurgi Serbuk kemudian digunakan untuk kompaksi dan proses sintering pada suhu yang berbeza (430˚C-594˚C). Keputusan boleh mempunyai implikasi penting dalam memproses CNTs yang diperkuatkan komposit matriks logam di luar angkasa, otomotif, industri elektronik dan secara umum. Dalam menyiasat mekanikal gabungan dan keadaan sintering,morphologi dan peningkatan sifat mekanik dibandingkan dengan aluminium asli dan Al-2wt% CNTs dalam proses yang sama.

Kata kunci: Mekanikal gabungan, matrik Al CNTs, Serbuk Metalurgi.

**OPTIMIZATION OF MILLING CONDITIONS AND SINTERING PROCESSES IN AL-CNTs ADVANCED NANOCOMPOSITE**

## ABSTRACT

Carbon nanotubes (CNTs) have recently emerged as materials with outstanding properties. Researchers have investigated their use as reinforcements in polymer and ceramic matrices. However, due to the anticipated fabrication difficulties, the research has been extended to use the material to reinforce metal matrices. In this project, planetary ball milling was used to disperse 2wt% MWCNTs in aluminum (Al) powder. Mechanical alloying (MA) was used to generate a homogenous distribution of 2wt% CNTs within Al powders. The effect of milling time (up to 40 hr) speed (200-250 rpm), ball size (10, 15 and 20 mm) and PCA (stearic acid, methanol) on the morphological development of the powders was investigated subsequently. Powder metallurgy was used for compaction and sintering process in different temperatures (430˚C-594˚C). The results can have important implications for the processing of CNTs reinforced metal matrix composites in aerospace, automotive, electronics industries, etc. Under the investigated mechanical alloying and sintering conditions, morphology and enhancement in mechanical properties were compared with pure aluminum and Al-2wt%CNTs in a same process history.

Keywords: Mechanical alloying, Al matrix CNTs, Powder metallurgy.

# CHAPTER 1

# INTRODUCTION

## 1.1 An overview of study

The interest in carbon nanotubes as super reinforcements for metallic matrixes has been growing considerably over the past few years, largely focusing on investigating their contribution to the enhancement of the mechanical performance of the composite. Since the landmark paper on carbon nanotubes (CNTs) by Iijima nineteen years ago (Iijima, 1991), researchers have demonstrated the exceptional potential and diversity of these materials. A single wall carbon nanotube, i.e. a nanotube made of only one graphite sheet rolled-up in cylinder, has a Young's modulus as high as 1.8TPa and a tensile strength as high as 63GPa (Popov, 2004), which is one to two orders of magnitude superior to the best known steels. Multiwall nanotubes display lower but still exceptional mechanical properties, and they are easier to synthesize. These superior mechanical properties combined with a low density generate several outlets for a composite reinforced by CNTs.

As in conventional composites, the orientation of the CNTs, homogeneity of the composite, nanotube matrix adhesion, its aspect ratio and the volume fraction of nanotubes are expected to have significant influences on the properties of the nanocomposite. Controlling such factors to obtain an exceptional composite is very challenging. A number of reviews have been published on the subject, including ones by Harris (2004), Carreno-Morelli (2006) and Curtin and Sheldon (2004). Aluminum has benefited from intense research efforts in this area, where CNTs are hoped to provide substantial improvements to its properties. One of the major obstacles to the effective use of carbon nanotubes as reinforcements in metal matrix composites is their agglomeration and poor distribution/dispersion within the metallic matrix. A number of researches have investigated the use of ball milling as a mechanical dispersion technique (Zhou et al., 2007; Bustamante et al., 2008). However, concerns about the possible damage and/or amorphization of the CNTs under the harsh milling conditions have been highlighted (Bustamante et al., 2008; Edtmaier et al., 2004), therefore optimization of milling conditions is deemed necessary.

Enhancements in mechanical properties due to CNTs inclusion (up to 10w%) have been highlighted (Kwon et al., 2008; Perez-Bustamante et al., 2008). The optimum CNTs content at which maximum enhancement is attained varies depending on mixing and preparation techniques. In general, the enhancements were much lower than expected (Deng et al., 2007; George et al., 2005). Most researchers have been cautious about processing CNTs composites at high temperatures so as to avoid CNTs from damage and possible adverse chemical interfacial reactions with the matrix. It has been argued, however, that the formation of $Al_4C_3$ phase should not be viewed as detrimental, since it can help to enhance the Al-CNTs bond and can lock the nanotubes in place and hence, contribute to the enhancement of the mechanical properties of the composites (Kwon et al., 2008 and Choi et al., 2008).

One way to produce composites with uniform distribution of CNTs within the metallic matrix is by first dispersing them in metal powders then subsequently consolidating these Al-CNTs composite powders. The ball milling process, although effective, normally results in strain hardening of the powders, and this can have implications on the final properties of the bulk composite. Therefore, the consolidation of heavily worked powders and their subsequent properties would be important from both a practical as well as a scientific standpoint.

CNTs have a strong tendency to form bundles due to their high aspect ratio and their van der Waals bonding (Kim et al., 2008). This extensive agglomeration, detrimental for composite mechanical properties, is a main issue when trying to use them as reinforcement (Robertson, 2004). Mechanical milling, a solid state high-energy ball milling process where particles are repeatedly fractured and welded (Suryanarayana, 2001), has been used successfully to disperse uniformly a variety of reinforcements within Al matrix (Fogagnolo et al., 2003), (Son et al., 2003; Ruiz-Navas et al., 2006). Furthermore, the mechanical properties of Al-CNTs composite made by mechanical milling would be further improved by Al grain refinement up to the nanoscale due to the intensive plastic deformation and by the incorporation and dispersion of the oxide layer initially present on the surface of Al powders (Suryanarayana, 2001). Also, studies of CNTs milling with metals, such as iron or magnesium for hydrogen storage (Wu et al., 2006) indicate accelerated CNTs damages. Only few research groups have investigated the dispersion of CNTs in an Al matrix by mechanical milling (Esawi and Morsi, 2007; George et al., 2005; Perez-Bustamante et al., 2008), and their investigation of the effect of milling on the nanotubes structure has been very limited.

### 1.2 Problem Statement

The present researchers believe that results for CNTs reinforced aluminum or even other metals should be viewed in light of the condition of the powders used to form the bulk samples and their subsequent process history (e.g. CIP, hot rolling and extrusion).

The points that have not been given too much attention so far in the literature are as follow:

- Consolidation and sintering processes of ball-milled Al-CNTs composite powders.

- Possible damage of CNTs as reinforcements in Al matrix composites by using high energy ball milling (as a mechanical size reduction technique) and high temperature processing or metal working.

- Interfacial reactions between the aluminum and CNTs.

## 1.3 Objectives

The measurable objectives of this project are:

a) To determine the effect of different ball milling parameters and operating conditions (milling time, ball size, PCA and speed) in Al matrix to optimize impact energy, milling temperature and achieve particle size reduction with less contamination.

b) To study on role of powder metallurgy and sintering process conditions due to improve consolidation and sintering factors (pressure, temperature and holding time) for fabrication of Al-CNTs nanocomposite.

c) To characterize the morphological and mechanical properties of Al-CNTs through test.

## 1.4 Scope of Work

(a) To apply milling machine for mechanical alloying and powder preparation. This includes the shape and size investigation.

(b) To use vacuum hot press machine for Al matrix CNTs bulk fabrication with study on bulk density and porosity.

(c) Characterization of the raw and milled powder by using scanning electron microscopy (SEM), X-Ray diffraction (XRD), Raman spectroscopy, densimeter and laser particle analyzer.

## 1.5 Thesis outlines

Chapter 1: In this chapter the general knowledge and information about Al-CNTs, the previous works and difficulties for fabrication of this nanocomposite are discussed.

Chapter 2: In this chapter the possibility methodology of producing Al-CNTs nanocomposite by mechanical milling and powder metallurgy technique is studied, in which there are more details and data of preparation and fabrication methods that were chosen for Al matrix carbon nanotubes.

Chapter 3: In this chapter, all the raw materials and equipments including measurements, testing facility and also powder preparation and bulk fabrication information which lead this project to be done are available.

Chapter 4: Here the emphasis being on the structural evolution of Al-CNTs upon milling and vacuum hot press machine by morphology and mechanical characterization of milled mixtures and pellet samples with X-ray diffraction (XRD), raman spectroscopy, scanning electron microscopic (SEM), densimiter measurement, microhardness and three-point bending (Insron) machine.

Chapter 5: In this chapter the final analysis discussion of the effects of mechanical alloying and consolidation parameters of unreinforced and reinforced Al and aluminum milled 2wt% CNTs is done, continuously a short recommendation and ongoing researches in Al-CNTs area is mentioned.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Introduction

Literature reviews for two main scope of the thesis are presented in this chapter. The scopes covered are shown as below:

- Mechanical alloying (MA)
- Powder metallurgy

### 2.1.1 Mechanical alloying /milling process

The objectives of milling are to reduce the particle sizes (breaking down the material) mixing, blending and particle shaping. Here, the focus is only on the application of milling (ball milling) for fabrication of engineering materials via MA process. Benjamin (1992) has defined the MA process as a method for producing composite metal powders with a controlled fine microstructure. It occurs by the repeated fracturing and rewelding of a mixture of powder particles in a highly energetic ball mill. As originally carried out, the process required at least one fairly ductile metal (Al) to act as a host or binder. Other components can consist of other ductile metals, brittle metals, and intermetallic compounds or nonmetals and refractory compounds. The major process in MA for producing quality powders of alloys and compounds with well-controlled microstructure and morphology is the repeated welding, fracture, and rewelding of the reactant mixed powders.

It is critical to establish a balance between fracturing and cold welding in order to mechanically alloy successfully. Two techniques were proposed by Gilman and Benjamin (1983) to reduce cold welding and promote fracturing. The first technique was to modify the surface of the deforming particles by adding a suitable processing control agent (PCA), (wet milling) that impedes the clean metal contact necessary for cold welding. The second technique was to modify the deformation mode of the powder particles so that they are able to deform to the large compressive strains necessary for flattening and cold welding. Cooling the mill chamber is an approach to accelerate the fracture and establishment of steady-state processing due to the effect of milling temperature (White, 1979). It should be emphasized that milling the powders of certain metal which will be cold-weld easily (Al) with an organic agent (PCA), (Rairden and Habesch, 1981) may lead to an undesired reaction between the PCA and the milled powders. Ductile/ductile components exhibit flattening and cold-welding into a lamellar structure which after sufficient repeated fracture and rewelding form homogeneous equiaxed particles.

Whenever two grinding balls collide, a small amount of powder is trapped in between them. Typically, around 1000 particles with an aggregate weight of about 0.2mg are trapped during each collision. During this process, the powder morphology can be modified. For the soft powders like aluminum particles, the flattened layers overlap and form cold welds. This leads to formation of layered composite powder particles consisting of various combinations of the starting ingredients. The work-hardened element or composite powder particles may fracture at the same time. These competing events of cold welding (with plastic deformation and agglomeration) and fracturing (size reduction) continue repeatedly throughout the milling period. Eventually, a refined and homogenized microstructure is obtained, and the composition of the powder particles is the same as the proportion of the starting constituent powders. Along with the cold welding event described above, some powder may also coat the grinding medium and/or the inner walls of the container. A thin layer of the coating is beneficial in preventing wear-and-tear of the grinding medium and also in preventing contamination of the milled powder with the debris.

The MA process is affected by several factors that are playing very important roles in the fabrication of homogeneous materials. It is well known that the properties of the milled powders of the final product, such as the particle size distribution, morphology and the degree of disorder, depend on the milling conditions and, as such, the more complete control and monitoring of the milling conditions, the better end product is obtained.

**2.2 Planetary ball mill**

In this type of mill, the milling media contains considerably high energy, as the milling stock and balls come off the inner wall of the vial (milling bowl) and the effective centrifugal force can reaches up to twenty times of gravitational acceleration. The centrifugal forces caused by the rotation of the supporting disc and autonomous turning of the vial act on the milling charge (balls and powders). Since the turning directions of the supporting disc and the vial are opposite, the centrifugal forces alternately are synchronized and opposite. Therefore, the milling media and the charged powders alternatively roll on the inner wall of the vial, and are lifted and thrown off across the bowl at high speed, as schematically presented in Fig. 2.1.

One advantage of this type of mill is the ease of handling the vials (45 ml to 500 ml in volume) inside the glove box.

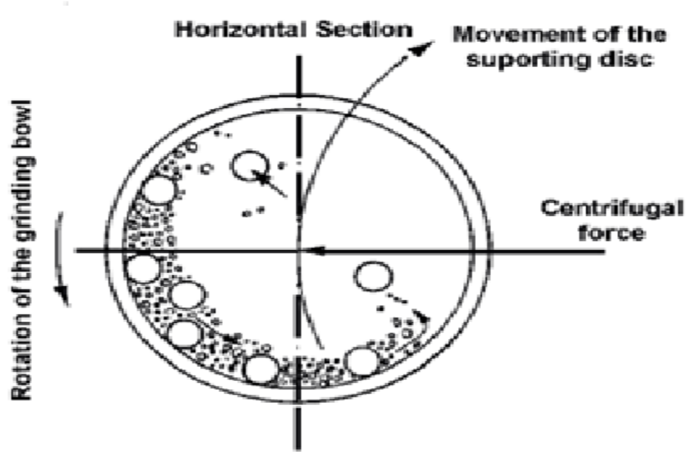


**Figure 2.1** Schematic drawing of a high-energy planetary ball mill.

The powder particles are repeatedly cold welded by the colliding balls as shown in Fig. 2.2 (Schultz, 1998). Thus, the composite powder particles with a characteristically layered microstructure (El-Eskandarany et al., 1992) are formed.

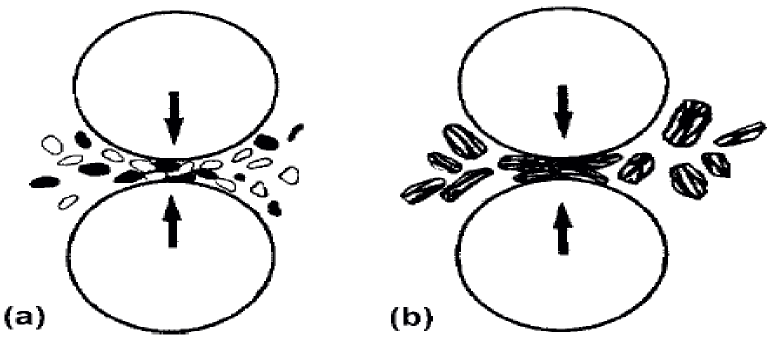


**Figure 2.2** Schematic presentation of the mechanical alloying process: (a) at the beginning of the milling and (b) after some collision events (Schultz, 1998).

### 2.2.1 Preparation of metal matrix nanocomposites by mechanical alloying

The field of nanocomposites has recently attracted considerable attention as researchers strive to enhance composite properties and extend their utility by using nanoscale reinforcements instead of the more conventional particulate-filled composites (Novak, 1993). While smaller reinforcements have a better reinforcing effect than larger ones, applying the ball-milling technique for composite fabrications provide the following merits:

• Since ball milling is processed at room temperature, the disadvantages of the liquid metallurgy method for producing undesirable materials can be avoided.

• Moreover, the ball-milling process can produce homogeneous nanocomposite powders instead of the more conventional coarse particulate-filled composites.

Metal matrix composites (MMC) are an engineered combination of two or more materials in which tailored properties are achieved by bringing the combined advantages of both reinforcement and metallic matrix into full play, which give a rather high degree of freedom in material design (Novak, 1993). Structurally, reinforcements may be in the form of continuous fibers, short fibers, particles, or whiskers, and early interest in developing these materials was fueled by their potential for applications in aerospace, naval and automotive structures. There are various methods employed in fabricating aluminum matrix composites using different processing parameters, such as melt process, powder metallurgy (PM), hot or cold pressing.

### 2.2.2 Fabrication methods of the nanoscale milled powders

Consolidation of milled powders is an important step for reliable and reproducible determination of the physical/mechanical properties of the fabricated materials and is required for most industrial applications (He and Ma, 1996; El-Eskandarany et al., 1998). Consolidation of the ball milled powders into bulk, full density compaction while retaining nanoscale grain size is a major challenge (He and Ma, 1996). Many sintering techniques, e.g., hot pressing (Nash et al., 1992) and hot extrusion (Hwang et al., 1992), sintering forging (Jain and Christman, 1994) and HIP-ing (Oehring et al., 1995) have been employed to consolidate the mechanically alloyed powder.

## 2.3 Powder metallurgy / powder compaction

Powder metallurgy is a forming and fabrication technique consisting of three major processing stages. First, the primary material is physically powdered, divided into many small individual particles. Next, the powder is passed through a die to produce a weakly cohesive structure very near to the dimensions of the part ultimately to be manufactured. Also, in order to attain the same compression ratio across more complex pieces, it is often necessary to use lower punches as well as an upper punch. Finally, the end part is formed by applying pressure, high temperature, long setting times (during which self-welding occurs), or any combination thereof. Main technique used to form and consolidate the powder is known as sintering. Among the various metal working technologies, powder metallurgy is the most diverse manufacturing approach. One attraction of powder metallurgy (PM) is the ability to fabricate high quality, complex parts to close tolerances in an economical manner. In essence, PM converts Al powders with specific attributes of size, shape, and packing into a strong, precise, high performance shape. Key steps include the shaping or compaction of the powder and then subsequent thermal bonding of the particles by sintering. The process can be effectively automated with low relative energy consumption, high material utilization and low capital costs. These characteristics make PM well aligned with the current concerns about productivity, energy, and raw material.

Consequently, PM process has become and replacing traditional Al forming operations. Further, powder metallurgy is a flexible manufacturing process capable of delivering a wide range of new materials, microstructures, and properties. That creates several unique and nice applications for PM in composites. The three main steps in powder metallurgy process are illustrated in Fig. 2.3.

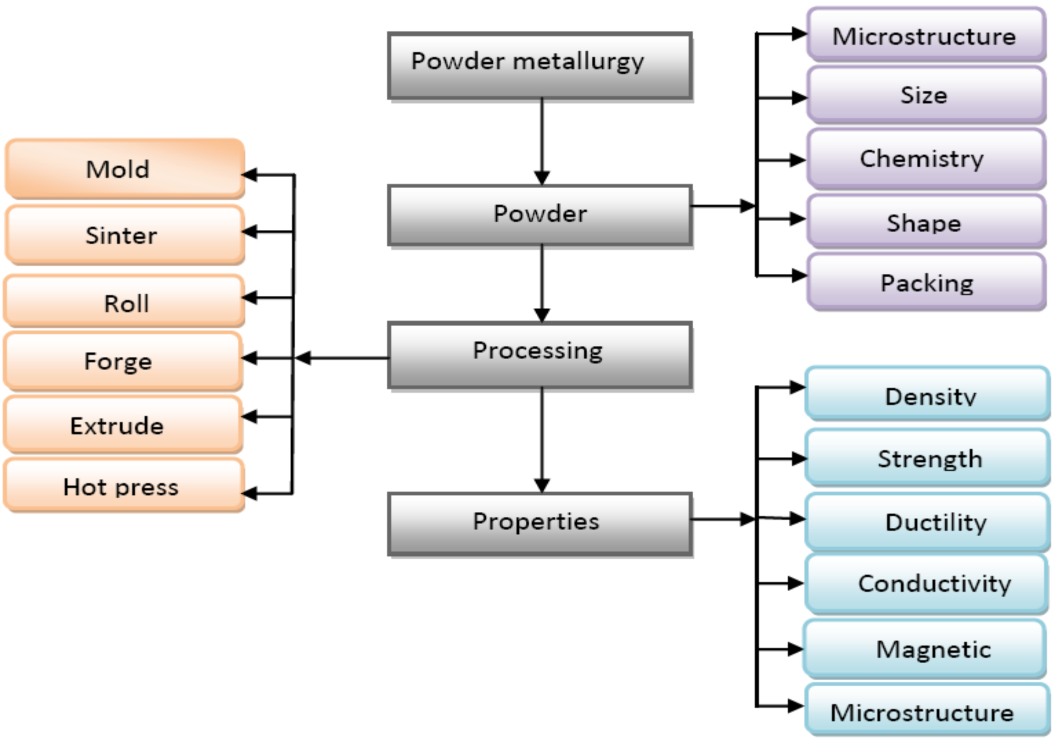


**Figure 2.3** The conceptual flow for powder metallurgy from the powder through the processing to the final product (German, 2005).

### 2.3.1 Thermo-mechanical processing

Vacuum Hot pressing is a process used to consolidate Al powder compacts to full density with controlled microstructures. Hot pressing has evolved as an alternative route to sintering to achieve high densification.

In vacuum hot pressing, there are three degrees of freedom in terms of varying the temperature, holding time and pressure during processing. The process involves the simultaneous application of pressure and heat to a ‘green’ component. Pressure is applied dynamically to the heated component in two opposing directions along a single axis. A vacuum or controlled atmosphere is required to prevent oxidation. During hot pressing, a mold provides the component with the desired shape. Heat is applied indirectly (convection or radiation).

Vacuum hot press machine provides the minimized condition of undesirable reactions between the Aluminium and oxygen, CNTs and the aluminium matrix. The simultaneous annealing of the strain-hardened powders is another advantage.

### 2.3.2 Sintering

Sintering is a consolidation method to compact loose powders to a bulk component. During this process, particles are bonded together and annihilated the voids, resulting in dense structures with complex shapes. This is followed by heating the green body (unsintered) at lower temperatures to burn off the binder and sintering at high temperature. Aluminum could be fabricated to complex shapes using this technique as well.

A schematic drawing of how particles fuse together by application of thermal energy is shown in Fig. 2.4 such process is dependent on diffusion of atoms that combine distinct powder particles into one cohesive material. Fusion occurs well below the melting point of the material, but at a temperature sufficiently high enough to allow an acceptable rate of diffusion to occur, usually at greater than one-half of the melting point on a Kelvin scale. The powder particles are thus compacted together thereby forming a compacted mass of powder particles that resembles a close packed crystal structure, with the powder particles being analogous to the atoms of the crystal.

Among the advantages of sintering process are:

- The purity of the material is guaranteed due to very few processing steps.
- The processing can be done in controlled environment.
- Easy to achieve complex shapes.

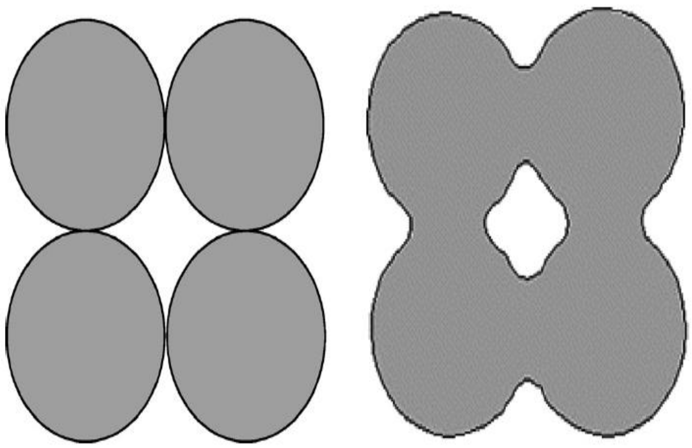

**Figure 2.4** A schematic showing individual particles fusing together and annihilating the voids.

## 2.4 Summary:

The major obstacles to the effective use of carbon nanotubes as reinforcements in aluminium matrix composites is their possible damage within the Al matrix, so in this project, effect of ball milling as a technique to minimize these problems with optimization of milling conditions (speed, time, ball size, ball no, PCA) regarding to size and morphology of aluminium-2wt% CNT are investigated.

The fabrication method for producing Al-CNTs composites is by first dispersing CNTs in Al powders then subsequently consolidation the Al-CNTs composite powders. The consolidation of ball-milled Al-CNTs composite powders is discussed in chapter 3 and 4 by using mechanical alloying and vacuum hot press machine under varied temperatures, holding time and pressures to identify the optimum temperature that leads to enhanced properties without resulting in excessive carbide formation in Al-CNTs composites to evaluate the mechanical response of the composites (both annealed and un-annealed) using mechanical testing.

# Chapter 3

# EXPERIMENTAL MATERIALS AND METHODOLOGY

## 3.1 Introduction

This chapter discussed about methodology used in this thesis and divided into 3 parts:

- To optimize milling parameters for preparation of aluminum matrix and Al-CNTs Powder.
- To modify parameters and variables sintering process for compaction of aluminum and Al-CNTs.
- To characterize the mechanical properties of aluminum, and Al-CNTs that were prepared in milling and vacuum hot press machines.

## 3.2 Experimental materials

In this section aluminum powder (matrix), MWCNTs (reinforcement), stearic acid and methanol (PCA) are shortly discussed.

Aluminum powder used was from Khorasan powder metallurgy, Iran with a purity >99%. The particle size is 90%, 31.97µm and is flaky in shape.

Multi-walled carbon nanotubes (MWCNTs) used was from Shenzhen, China with average particle size of 10-20 nm external average diameters, 5–15µm length and 95-98% purity.

In this study stearic acid ($C_{18}H_{36}O_2$ or $CH_3(CH_2)_{16}COOH$) and also methanol ($CH_3OH$) with purity 99% from Fisher chemicals and Merck with purity 99.99% used as process control gent separately in aluminum and Al-CNTs to avoid cold welding.

### 3.3 Characterization procedures

Laser particle analyzer, SEM, XRD, Raman and TGA are included in this part.

#### 3.3.1 Laser particle size analyzer

Particle size analyzer measures the dimensions of a particle. The size of a particle depends on the measurement technique. In this research, powder particle size and distributions were determined using a CILAS 1190 instrument (Fig. 3.1) with a measurement range from 0.04 to 2,500μm.


**Figure 3.1** CILAS 1190 particle size analyzer.

### 3.3.2 Powder morphology observation

A field emission scanning electron microscope (FESEM ) model LEO supra 55 with up to 40nA probe current was used to observe the morphology and contamination (EDX) of the powder. The machine used during the study is shown as in Fig. 3.2.

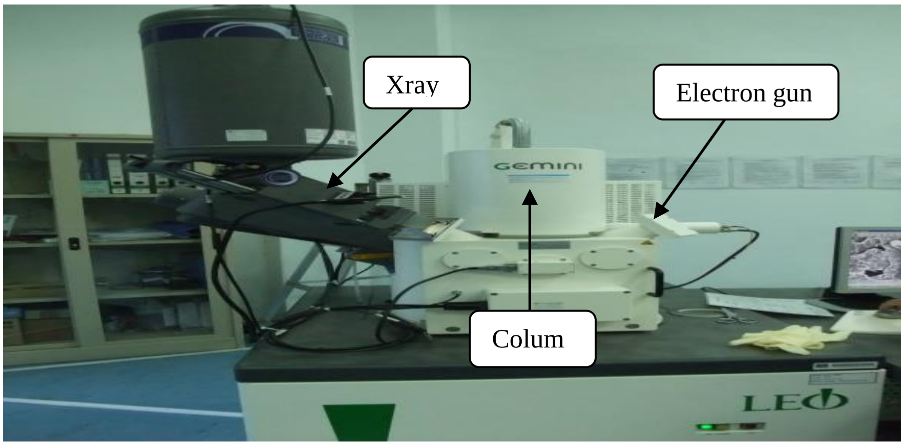


**Figure 3.2** Leo FE-SEM ultra high resolution with GEMINI column.

### 3.3.3 X-Ray diffraction analysis (XRD)

XRD characterization of the powders was performed with a Bruker type using $C_uK_\alpha$ radiation at 4.8 kw as shown in Fig. 3.3.

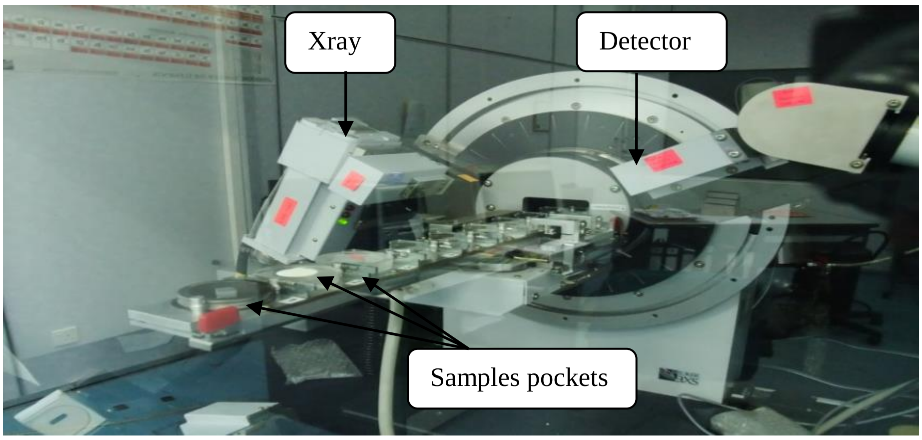


**Figure 3.3** Bruker X-Ray diffraction (XRD).

### 3.3.4 Raman spectroscopy analyzer

Raman spectra were recorded with a Renishaw Invia Raman spectrometer with an argon laser light (514 nm) to prove quality of CNTs (Fig. 3.4). To reach stronger reinforcement product, minimize damage of CNTs during milling and homogenous composite powder the quality of MWCNTs is important.

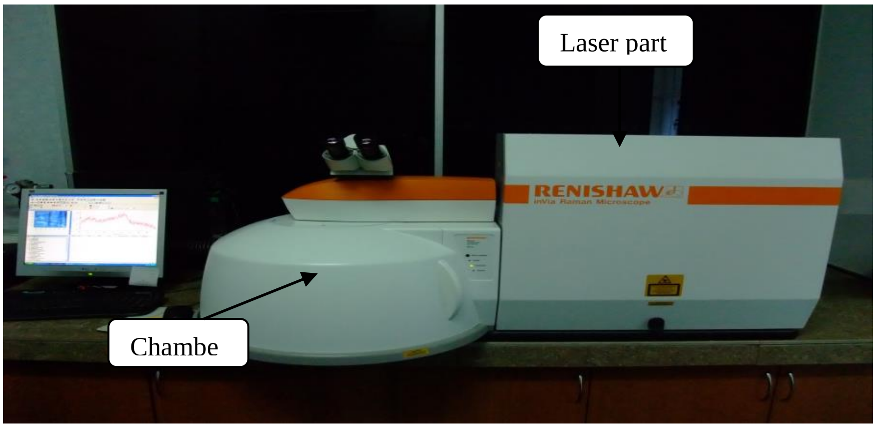


**Figure 3.4** Renishaw Invia raman spectrometer.

### 3.2.5 TG/DTA analyzer

Thermal and oxidation stability of materials and phase transformation during mechanical milling process were studied with thermo gravimetric analysis (TG/DTA SII 6000 Series) as shown in Fig. 3.5.

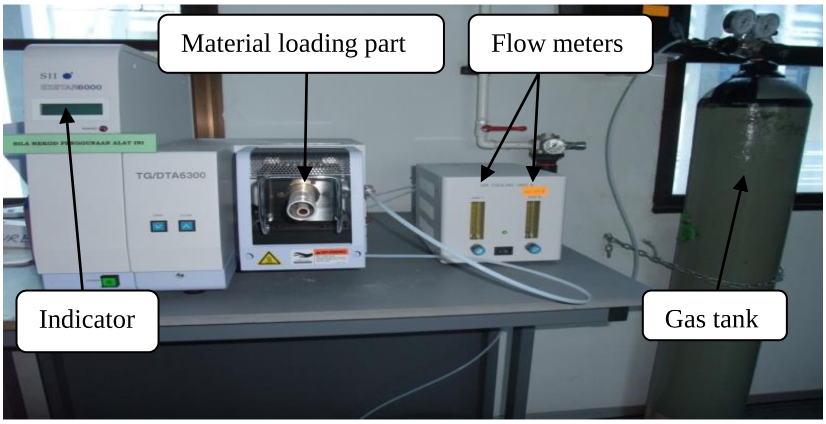


**Figure 3.5** Thermogravimetric analyzer (TG/DTA SII 6000 Series).

### 3.4 Composite preparation and experimental procedure

The essential step in preparing aluminum and Al-CNTs process can be divided into 2 stages:

(1) Mixing/mechanical alloying

(2) Compaction and/or sintering

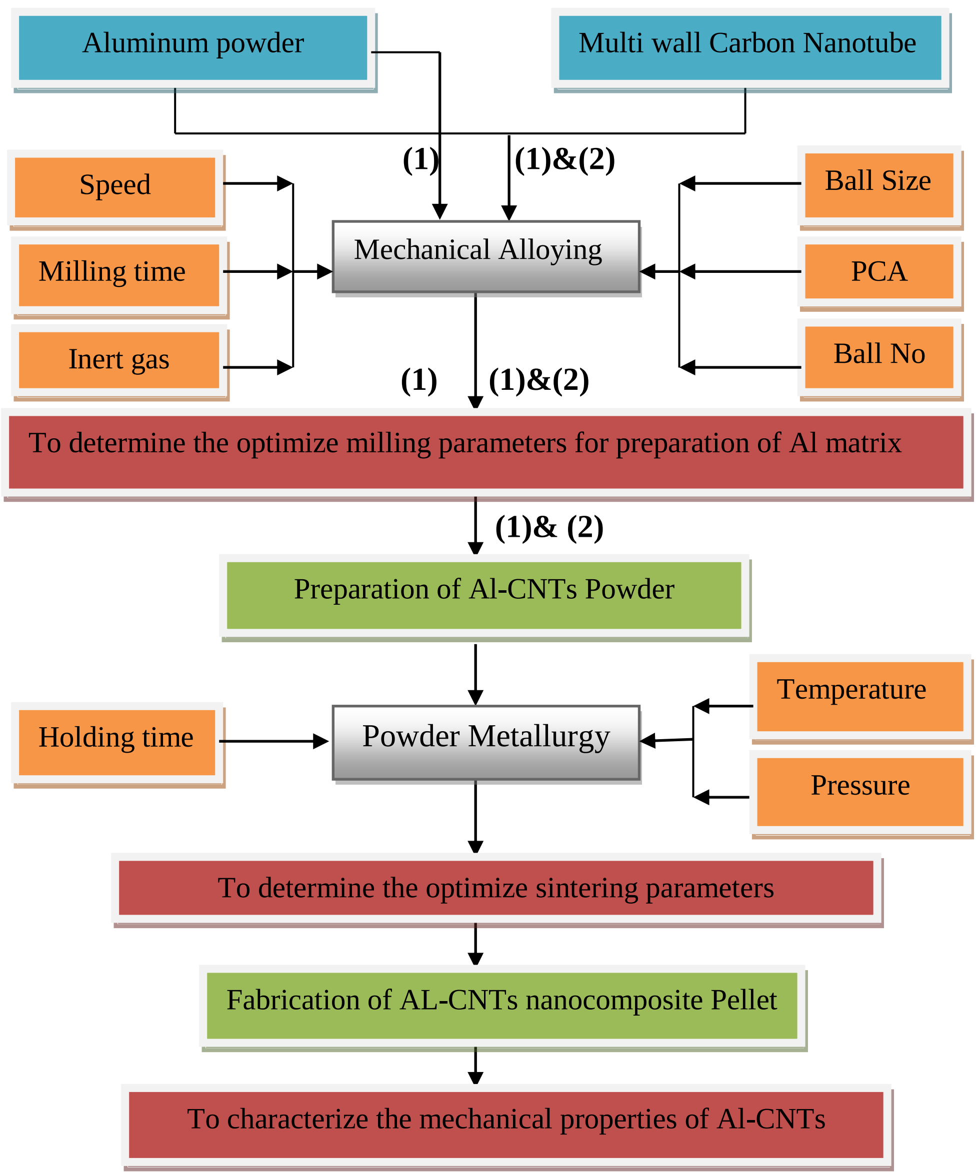


**Figure 3.6** Essential steps in the preparation of aluminum and Al-CNTs.

### 3.4.1 Mechanical alloying

First of all pure aluminum was ball milled using 500ml stainless steel mixing planetary ball mill Retsch 400M (110mm in inner diameter and 110mm in height, Fig. 3.7).

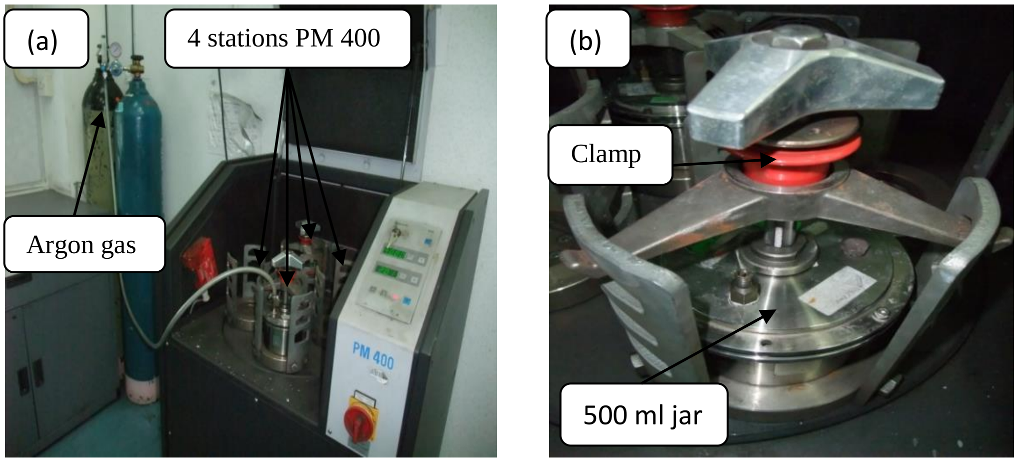


**Figure 3.7** (a) Retsch PM400, (b) 500 ml jar and clamp.

The jars were ran under argon gas then were agitated with 3 different stainless steel milling balls size (10, 15 and 20mm) at two different rotation speed (200 and 250rpm), two different ball number (31 and 100) for varying milling times up to 40 hr. 0,1,2, and 3 wt.% of stearic acid [$CH_3(CH_2)_{16}COOH$] and 3.33 ml methanol [$CH_3OH$] were added as a process control agent (PCA) separately and giving a ball to powder weight ratio (B/P) 10:1 to reach a fine and small powder particle size (~10µm) regarding to powder morphology and to minimize cold welding of the aluminum particles. In addition, the PCA was used to prevent powders from sticking to the balls and the jar wall (step 1).